

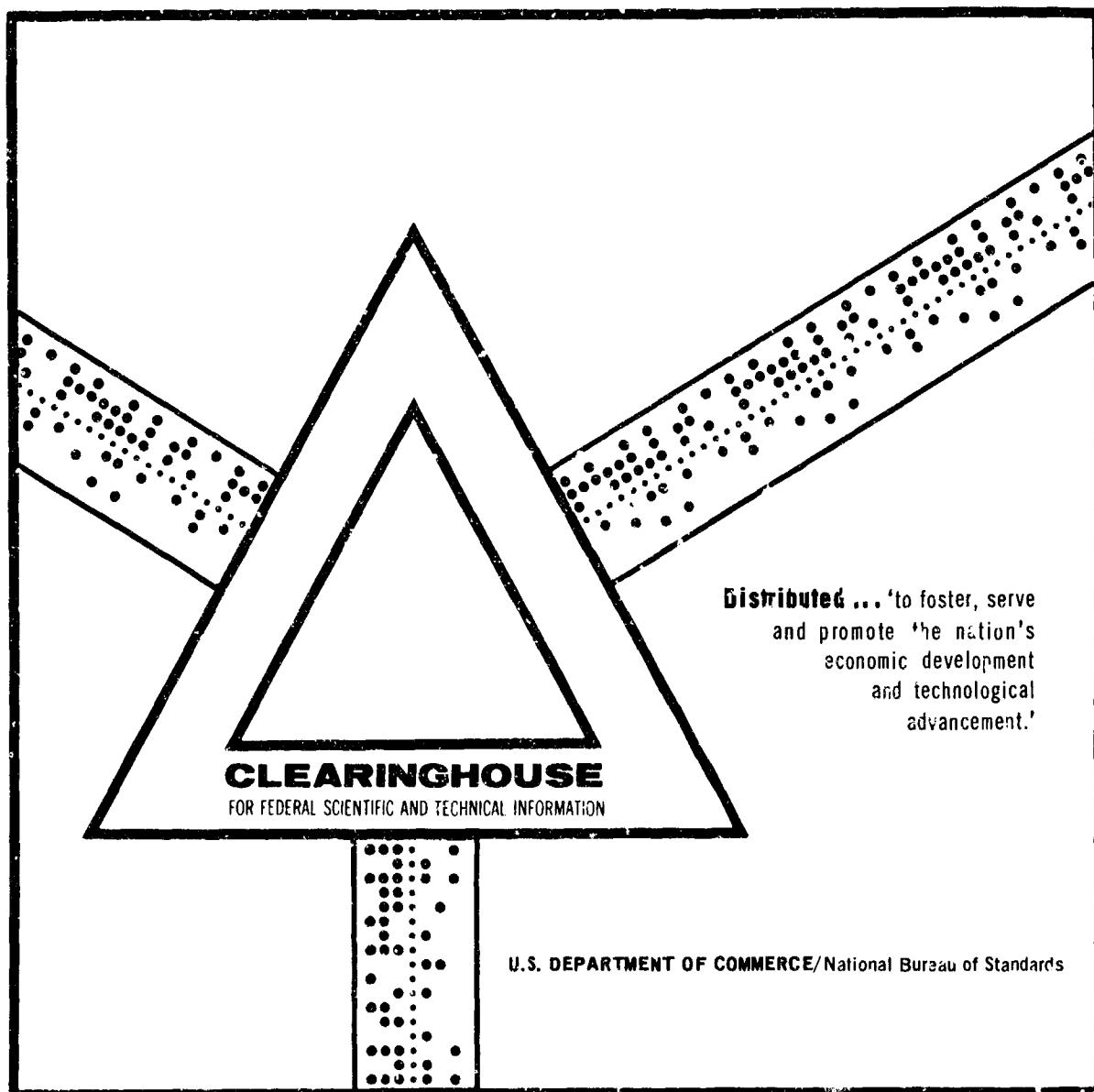
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IMPACT SENSITIVITY OF LEAD AZIDE IN VARIOUS
LIQUIDS WITH DIFFERENT DEGREES OF CONFINEMENT

Louis Avrami, et al

Picatinny Arsenal
Dover, New Jersey

November 1969



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TECHNICAL REPORT 3965

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LOUIS AVRAMI
NATHANIEL PALMER

NOVEMBER 1969

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Technical Report 3965

IMPACT SENSITIVITY OF LEAD AZIDE IN VARIOUS LIQUIDS
WITH DIFFERENT DEGREES OF CONFINEMENT

by

Louis Avrami
Nathaniel Palmer

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Explosives Laboratory
Feltman Research Laboratories
Picatinny Arsenal
Dover, New Jersey

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ABSTRACT

The work described in this report was conducted to determine the sensitivity of unconfined mixtures of lead azide with various liquids as measured with an impact machine. The test was developed by so modifying the standard Picatinny Arsenal impact test that the confinement of the sample was reduced; a well was introduced to retain the sample in relatively large amounts of liquid during impact.

The results indicate that dry, unconfined lead azide is initiated by impact at lower drop heights than the same material confined in the standard impact-test fixture. The reason for this apparent increase in impact sensitivity is not clear; it may be attributed to some uncontrolled experimental parameter. Unconfined mixtures of lead azide and water, lead azide and alcohol, lead azide and water-alcohol, and lead azide and Freon TF were found to be less sensitive to impact than dry lead azide.

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INTRODUCTION

Several recent reports (Ref 1 through 4) indicate that, as measured on the Picatinny Arsenal impact machine in a confined state, mixtures of lead azide with various liquids are more impact sensitive than dry lead azide. Also related to these results is an earlier report (Ref 5) which states as well that, as measured on the Naval Ordnance Laboratory impact tester, wet lead azide is more impact sensitive than the dry substance.

EXPERIMENTAL PROCEDURES

Sample Preparation

Lead azide precipitated in the presence of carboxymethylcellulose was used throughout this study. The material was prepared by Joliet Arsenal and described as Lot JA-4-61. As received, the lead azide was immersed in an alcohol solution. Batches weighing 200-250 grams were placed on a Büchner funnel and washed with five portions of a mixture of 1:1, by weight, of triple-distilled water and 95% ethyl alcohol. Suction was applied for an hour, and then each batch was dried for 24 hours in a vacuum oven at 60°C. Upon removal from the oven, each sample was placed in a conductive rubber container which was kept sealed until required for use.

Impact Sensitivity Determinations

Picatinny Arsenal Method

The Picatinny Arsenal impact machine was used with the standard operating procedure (Ref 6), applying 1- and 2-kilogram drop weights, to determine the impact sensitivity of dry lead azide. A different procedure was used for mixtures of lead azide with Freon TF: The lead azide was put into a container holding Freon TF, a volumetric sample was scooped from the mixture and loaded into the die cup, and then additional Freon TF was added drop by drop from a burette until the die cup was filled. The subsequent steps followed the procedure described in a previous report (Ref 1).

Impact Test Methods for Unconfined Samples

Several modifications of the Picatinny Arsenal machine were introduced to achieve different degrees of

confinement of the sample at the time of impact. To reduce the confinement, the sample was located on a base plate or a flat surface so that the drop of the striker or anvil would result in the impact of one flat steel surface upon another. In order to hold a liquid, solid steel cylinders 2" in diameter (D) and 1 1/2" in height were machined, and wells of different sizes were reamed out. Their measurements were 1" D x 3/8" deep, 1" D x 1" deep, and 1 3/4" D x 1" deep (Fig 1), and they were tempered to about the same hardness as the standard anvils (Fig 2). Two different anvils were used. The standard anvil was employed with the 3/8" deep well, and an extension of 2" was added to the shaft for the deeper wells. The other anvil was a 3/8" D steel punch about 3" long (Fig 1, 3). The selection of the 3/8" D punch was based on the diameter of the standard anvil's striking surface, which was formed like that of a truncated cone. It was realized that the standard anvil would displace more liquid than a straight punch when dropped into a filled well.

The first series of tests with the different liquids utilized the 1" D x 3/8" deep well with the standard anvil (Fig 1), which, when the tests were repeated, was replaced by the 3/8" D punch.

The second series of tests duplicated the first, but used a 1" D x 1" deep well. Due to the depth of the well, the anvil with the extension was applied. In the third series of tests, the 1 3/4" D x 1" deep well was used.

An additional modification was incorporated for the last series of tests. The 1 3/4" D x 1" deep well was used with a guide slipped over the top of the steel cylinder (Fig 3). The guide had a hole in the center, which permitted the 3/8" punch to rest on the explosive in the well in a perpendicular position (Fig 4). The test was performed by dropping a weight onto the punch (Fig 5).

The tests were conducted with different liquids and with the liquids at two different levels: One level was the maximum practicable one (1" deep), while the other was just above the surface of the explosive (~ 1/8").

With the modified impact machine, tests were conducted on dry lead azide, using both the anvil and the punch. They were followed by a second series of tests which, as a function of depth and width of the well, were performed on lead azide in different liquids, i.e., Freon TF, tap

water, 100% ethyl alcohol and a 50/50 mixture by weight of water and alcohol.

For the tests with the lower degree of confinement, each dry lead azide sample consisted of 25-30 milligrams. In all of the tests performed in a liquid medium, the weight of the explosive was 100 milligrams. The addition of the liquid to the explosive in the well tended to level out the explosive at the bottom of the well. The larger weight of the explosive in the liquid medium was necessary to ensure a reasonable depth of explosive in the well. The depth of the lead azide was approximately the same as the depth of the cup on the standard impact tester.

Each test was conducted to obtain a curve of percent "fires" as a function of the distance traveled by the drop weight. Twenty samples were tested for each distance of fall, and normally 6-10 distances (i.e., initial heights of the drop weight) were selected to obtain the range from "fires" (F) to "no fires" (NF). The minimum interval between different drop heights was one inch, the maximum height that could be obtained with the modifications 33 inches. In most of the tests, the ambient temperature and relative humidity were maintained within $\pm 4^{\circ}\text{F}$ and $\pm 3\%$ RH.

RESULTS

Six series of impact tests (totalling 38 tests) were conducted. All but 3 of these 38 tests were performed on unconfined samples.

In Table 1 the results are tabulated for dry lead azide obtained with the following three methods: (a) the standard Picatinny Arsenal test, (b) the unconfined test with the standard anvil, and (c) the unconfined test with the punch. The tests were performed with both the 1- and 2-kilogram drop weights. Tables 2, 3, and 4 record the results for the mixtures of lead azide with various liquids, obtained in the unconfined test as a function of the depth and width of the well. Table 2 shows the results obtained with the 1" D x 3/8" deep well, Table 3 those with the 1" D x 1" one, and Table 4 those with the 1 3/4" D x 1" one. All the results were obtained with the 2-kilogram drop weight, using the standard anvil or the punch.

Table 5 lists the results obtained with the 1 3/4" D x 1" deep well and the punch with guide. After the

explosive and liquid were placed in the well, the guide and the punch were installed, and the 2-kilogram weight with the standard anvil dropped. The tests were performed with the level of the specific liquid both at the maximum height and at the height just above the surface of the explosive, as described in the preceding section.

Evaluation of the data indicated that, using the above methods, the unconfined mixtures of lead azide with Freon TF, water, alcohol, or water-alcohol were less sensitive than the unconfined dry substance.

Some of the characteristics of the events that were recorded as "fires" are noteworthy. The audible response produced by a "fire" was, for the unconfined dry lead azide, noticeably lower than that produced by standard impact test explosions. Also, after a "fire" some lead metal usually fused to the striking surface of the base plate, and if these items were not cleaned or replaced, the samples showed an enhanced sensitivity to impact. This may have contributed to the observed greater sensitivity of unconfined lead azide as compared with that obtained with the standard Picatinny Arsenal impact test, but the possibility was not systematically investigated.

No discernible sound of explosion was heard during any of the "fires" during impact tests on the lead azide-liquid mixtures, but other evidence of initiation was noted: a lead deposit was found either on the anvil striking surface or on the bottom of the well; the liquid also became murky.

The sound of the "fires" which occurred during the tests was markedly different in the tests using the guide with the punch resting on the explosive: they were the most audible of the tests using liquids. Only in this series were sparks seen in some of the "fires". The responses in Freon TF were the most audible of the different liquids. Minute lead deposits also were observed when reaction occurred.

DISCUSSION

The assumption was made that for a great many operations the lead azide would be handled in mixtures with different liquids and in what could be considered a relatively unconfined state.

The data were subjected to statistical analysis by the method proposed by Kemmey (Ref 7) and used by Avrami and Jackson (Ref 1). That method combines the χ^2 (chi-square) or goodness-of-fit test with the Karber test to provide a means to rate the materials in terms of relative sensitivity. Briefly, when the χ^2 test indicates that two materials are significantly different, the Karber method is useful in arranging them according to sensitivity. In the χ^2 test the 95% level of confidence was chosen, and the degrees of freedom correspond to the number of drop heights.

Comparisons between pairs of samples could not be obtained if the measurements had not been made at the same heights. Even under these circumstances, however, the available computer program still permitted the Karber statistical analysis to be performed and also gave a graphical plot of the percent firing versus drop heights. In most cases where the χ^2 test could not be applied, differences in sample behavior were shown on the graphs, and the mean critical height and standard deviation from the Karber analysis indicated the relative order of sensitivity.

The results listed in Table 1 were subjected to statistical analysis, and graphical plots were made for both the 1- and 2-kilogram drop weights. The analysis indicated that in both cases the curves for unconfined dry lead azide were statistically different from, and indicated also more sensitivity than, the Picatinny Arsenal impact test curve (Fig 6 and 7). However, that analysis assumed that all the testing conditions were identical. The increase in sensitivity might be due to an increase in the energy actually imparted to the sample in each test. It was noted that this set of seemingly anomalous data was obtained in experiments in which lead deposits were allowed to accumulate on the striking surfaces of the impact fixtures. Earlier work on barium azide (Ref 8) has shown that metallic barium can sensitize the azide, and it is possible that the metallic lead had the same effect here. However, this possibility was not studied further, and so this conclusion must be considered speculative.

The results shown in Tables 2, 3, and 4 could not be analyzed according to the above method, since the height limitation of 33" did not permit a full curve to be obtained for any of the mixtures of lead azide with a liquid as a function of depth and width of the well. The

results are random and sporadic and the reactions infrequent. In 23 tests involving 3 wells and 4 liquids, only one test had 20% "fires" at 33", and 3 other tests had 15% at the same height. As the diameter and depth of the well were increased, fewer "fires" were obtained. As shown in Table 4, with a well 1 3/4" D x 1" deep, and using the anvil, only 3 "fires" were obtained from 560 samples tested using drop heights ranging from 9" to 33"; no "fires" occurred when 560 samples were tested with the punch.

The probable explanation of these data is as follows: When the falling weight either in the form of the anvil or the punch, hits the liquid surface, it acts as a piston, pushing liquid in front of it. Since the explosive was not confined, the liquid served as a lubricant which permitted the piston to disperse the explosive before the anvil or punch hit the bottom of the well.

Of all the modifications, the tests with the most confinement were performed with the guide and punch as shown in Figure 5. This forced the lead azide to be sandwiched between two flat steel surfaces in a liquid medium, and the response of the explosive was affected by the stimulus applied to the punch by the falling weight. This stage of the experiments was divided into two parts: One with the well filled, and the other with the liquid just above the surface of the explosives.

Although the χ^2 test could not be applied to the results obtained from both parts because the drop heights were not the same as those with dry lead azide, the Karber test did give the mean critical height and a graphical plot (Fig 8 and 9), so that a comparison could be made with the results for dry lead azide. Also included were the results obtained on the Picatinny Arsenal impact test machine for dry lead azide, and also for lead azide and Freon TF. In some cases, the analysis indicated that the distribution was not adequate to obtain a mean critical height. In such cases the curves in the graphs (Fig 6 and 7) are "best fits" to the data points.

The results indicate that under the unconfined conditions described above, the mixtures of lead azide and water, lead azide and ethyl alcohol, lead azide and 50/50 alcohol-water, and lead azide and Freon TF are less sensitive to impact than dry lead azide. Although the order of sensitivity for the mixtures with the different liquids was not duplicated when the well was partially filled, as

compared with a completely filled well, the sensitivity in both cases was reduced by a factor of almost three. In the modified test, the unconfined mixtures were less sensitive than dry lead azide or mixtures of lead azide and Freon TF as determined by the Picatinny Arsenal impact machine.

As an example of compilation of the test results reflecting the effects of Freon TF in all the tests--unconfined and standard--the pertinent data are shown in Table 6.

The test results obtained by Siele and associates (Ref 9) on the effects of relatively small amounts ($\sim 1\%$) of liquids, the results of Avrami and Jackson (Ref 1) with larger amounts of liquids ($\sim 28\%$), and the results given here, are in agreement in that the sensitivity of the mixtures of lead azide with various liquids increases as the confinement increases and as the rate and amount of stimulus increases. The maximum sensitivity for the mixtures dealt with in this report is achieved by the method used by Avrami and Jackson (Ref 1), since there the boundary conditions are fixed. The sensitivity is lowered as the confinement and the rate of stimulus application are reduced: A variation in any of these parameters will affect the sensitivity. In other words, a reduction in the confinement and in the rate of application of impact will reduce the probability of initiation of this type of lead azide if it is contained in a liquid medium. The possibility of an explosion occurring due to impact is, however, not precluded by the presence of the liquid.

ACKNOWLEDGEMENT

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TABLE I
Results of different impact test methods with lead azide (Lot JA4-61)

Drop Height in.	2 kg Drop Weight						1 kg Drop Weight					
	Standard P.A. Test			Unconfined w/Std Anvil			Unconfined w/3/8" Punch			Standard P.A. Test		
	F	NF	F	NF	F	NF	F	NF	F	NF	F	NF
1	0	0	20	0	20	0	0	20	0	0	20	0
2	20	4	16	0	20	3	17	0	20	1	19	0
3	0	6	14	3	17	5	15	0	19	1	19	0
4	19	7	13	5	15	7	7	1	16	4	16	0
5	18	10	10	13	10	7	4	16	4	16	4	16
6	15	10	10	10	9	11	4	16	4	16	4	16
7	9	11	11	9	8	12	0	9	11	4	16	0
8	14	6	16	4	10	10	0	9	11	4	16	0
9	16	4	19	1	18	2	0	12	8	8	12	0
10	15	4	20	0	19	1	0	16	4	10	10	0
11	20	0	0	0	20	0	0	2	18	3	12	8
12												
13												
14								2	18	0	11	9
15								3	17	0	11	9
16								11	9	0	11	9
17								16	4	0	12	8
18												
19												
20												
21												
22												
23												
24	E. 50	7.35+1.961	5.35+2.747	a	15.82+5.509	8.55+2.591	a	13.42+5.522	20.0	6	56%	56%
	Test No.	1	2	3	4	5	5	5	5	5	56%	56%
	R.H.	55%	60%	56%	55%	56%	56%	56%	56%	56%	56%	56%
	Temp.	76°F	77°F	76°F	77°F	76°F	77°F	76°F	77°F	76°F	77°F	76°F

^a Computer could not produce an accurate E.50.

Note: Lead azide was in a dry condition when used in these tests.

TABLE 2
Impact tests with unconfined lead azide in different liquids
using 1" D x 3/8" deep well and 2 kg drop weight

Drop Height in.	with standard P.A. anvil												with 3/8" punch													
	In			In			In			In			In			In			In			In				
	Dry	Water	Alcohol	50/50	Freon	Dry	Water	Alcohol	50/50	Freon	Dry	Water	Alcohol	50/50	Freon	Dry	Water	Alcohol	50/50	Freon	Dry	Water	Alcohol	50/50	Freon	
1	0	20				0	20				0	20				0	20				0	20				
2	4	16									3	17														
3	6	14									5	15														
4	7	13									13	7														
5	10	10									9	11														
6	10	10									8	12														
7	11	9									10	10														
8	14	6									10	10														
9	19	1									18	2														
10	20	0									19	1														
11											20	0														
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33																										
K.H.	60%	57%																								
Temp.	77°F	70°F																								
Test No.	2	8																								

Note: Dry lead azide results from Table 1.

TABLE 3
Impact tests with unconfined lead azide in different liquids
using 1" D x 1" deep well and 2 kg drop weight

Drop height in.	with standard envil (modified)						with 3/8" punch					
	In			In			In			In		
	Dry	F	NF	Water	Alcohol	50/50	Freon	Dry	Water	Alcohol	50/50	Freon
1	0	20						0	20			
2	4	16						3	17			
3	6	14						5	15			
4	7	13						13	7			
5	10	10						9	11			
6	10	10						8	12			
7	11	9						10	10			
8	14	6						18	2			
9	19	1		0	20	0	20	0	20	0	20	0
10	20	0						19	1			
11								20	0			
12												
15	0	20	0	20	0	20	0	20	0	20	0	20
18	0	20	0	20	0	20	0	20	0	20	0	20
21	0	20	1	19	0	20	0	20	2	18	1	19
24	1	19	1	19	1	19	0	20	1	19	1	19
27	1	19	0	20	1	19	0	20	2	18	1	19
30	0	20	0	20	0	20	0	20	2	18	2	18
33	0	20	1	19	2	18	0	20	3	17	4	16
H.H.	60	57	55	56	56	56	56	57	56	56	56	55
Temp.	77°F	74°F	75°F	78°F	72°F	76°F	78°F	76°F	78°F	76°F	74°F	75°F
Test No.	2	15	16	17	18	19	20	21	22	21	20	19

Note: Dry azide results from Table 1.

TABLE 4
Impact tests with unconfined lead azide in different liquids
using 1 3/4" D x 1" deep well and 2 kg drop weight

Height in.	With standard anvil (modified)												With 3/8" punch																	
	In Water						In Alcohol						In Freon						In Water						In Alcohol					
	Dry	F	NP	P	NP	F	NP	F	NP	P	NP	F	NP	P	NP	F	NP	P	NP	F	NP	P	NP	F	NP	P	NP			
1	0	20										0	20																	
2	4	16										3	17																	
3	6	14										5	15																	
4	7	13										13	7																	
5	10	20										9	11																	
6	10	10										8	12																	
7	11	9										10	10																	
8	6	14										10	10																	
9	19	1	0	20	0	20	0	20	0	20	0	18	2	0	20	0	20	0	20	0	20	0	20	0	20	0				
10	20	0										19	1																	
11												20	0																	
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R.H.	60%	53%	57%	57%	57%	68°F	70°F	70°F	69°F	69°F	69°F	76°F	69°F	55%	55%	56%	56%	56%	56%	56%	56%	56%	56%	56%	56%	56%	56%	56%		
Temp.	77°F	68°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F	72°F			
Test No.	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2		

Note: Dry lead azide results from Table 1.

TABLE 5
Comparison of impact tests with unconfined lead azide in different liquids
using 1 1/4" D x 1" wall, guide and punch (treating on explosive) with 2 kg drop weight

Drop weight, kg.	Unconfined w/ anvil		Unconfined w/punch		Well filled w/water		Well partly filled w/water		Well filled w/alcohol		Well partly filled w/alc.		Well filled w/50/50 filled w/50/50		Well partly filled w/Freon		Well partly filled w/Freon		P.A. test w/Freon	
	N	F	NF	V	HP	F	HP	F	NF	V	HP	F	NF	V	HP	F	NF	F	NF	
1	1	2	20															0	20	
2	1	4	16	6	22													11	0	
3	1	4	14	1	17													12	8	
4	1	4	11	5	15													18	2	
5	1	2	12	4	17													19	1	
6	1	2	10	9	11													20	0	
7	1	2	9	10	12													20	0	
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TABLE 6
Effect of Freon TF in impact tests with confined and unconfined lead azide with 2 kg drop weight

Note: Dry azide results from Table 1.

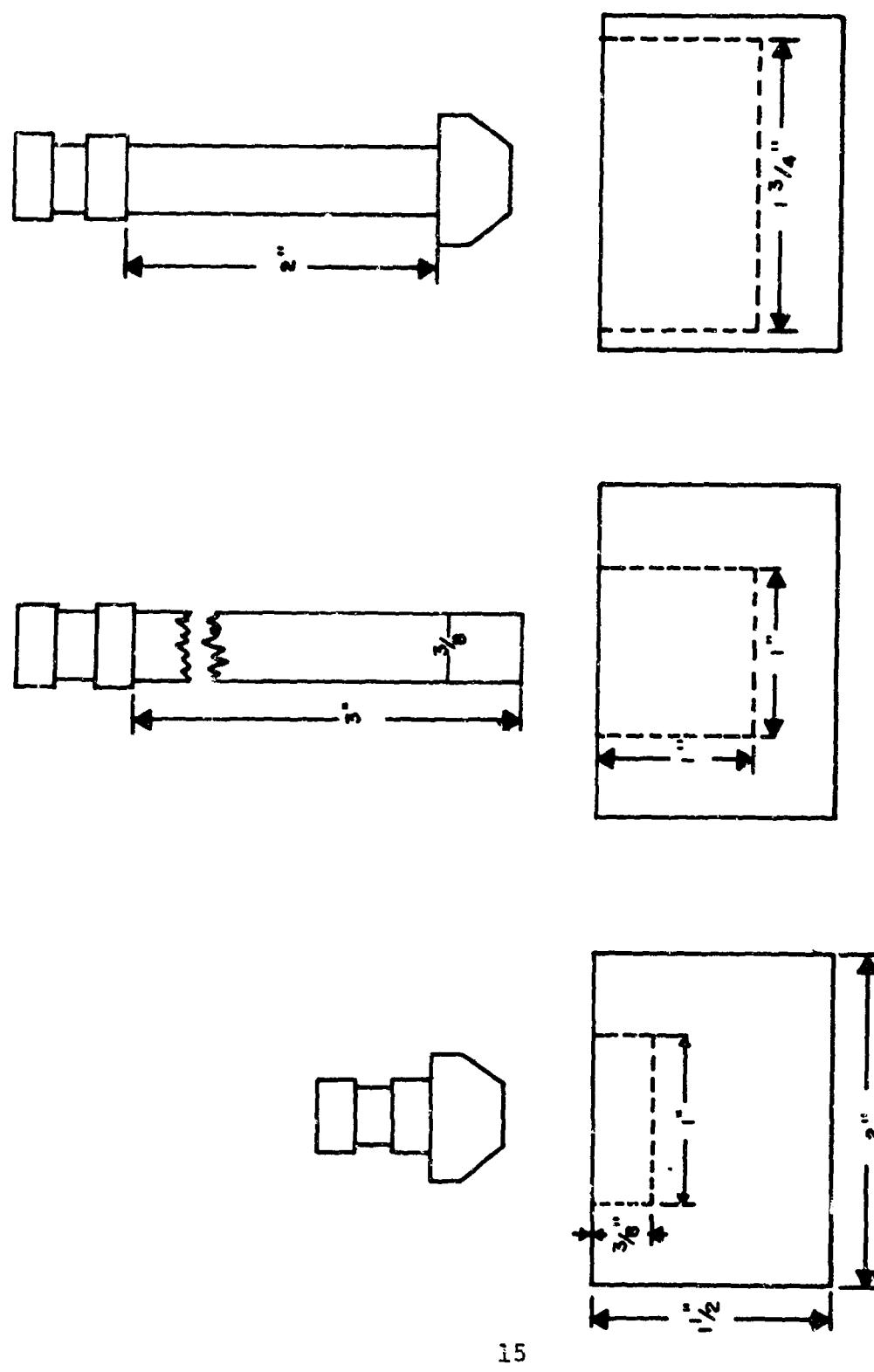


Fig 1 Drawings of wells, punch and anvils used in impact testing of unconfined lead azide in various liquids

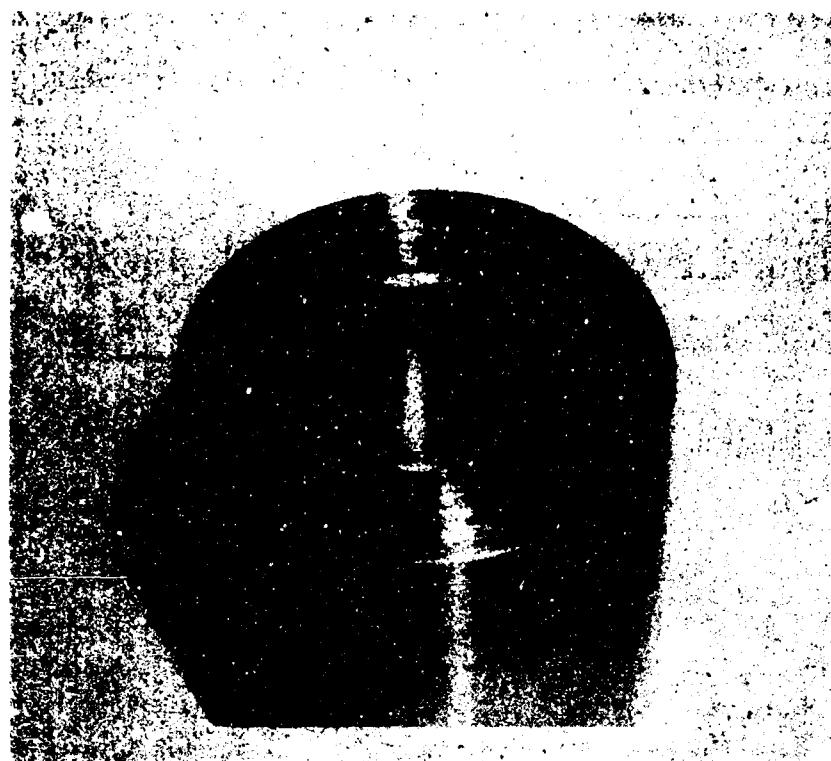


Fig 2 A 2" D x 1 1/2" H steel cylinder with
1" D x 1" deep well used to retain mix-
tures of lead azide and different liq-
uids for impact testing



Fig 3 Modified impact tester with 3/8" punch as anvil and 1" D x 1" deep well for unconfined testing



Fig 4 Modified impact tester with 1 3/4" D x 1" deep well with guide and punch (punch resting on explosive)

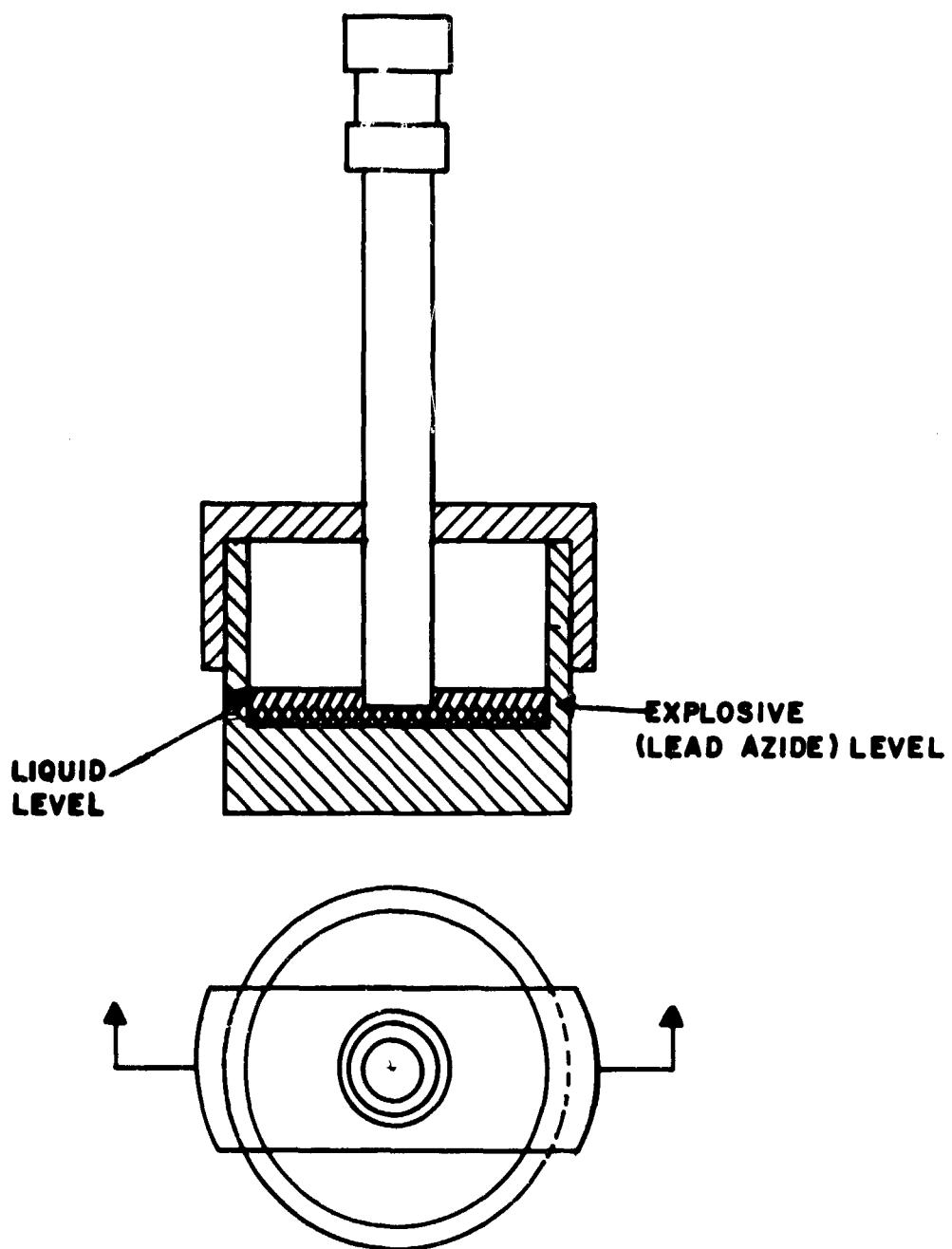


Fig 5 Sketch of 1 3/4" D x 1" deep well with punch and guide showing explosive and liquid levels

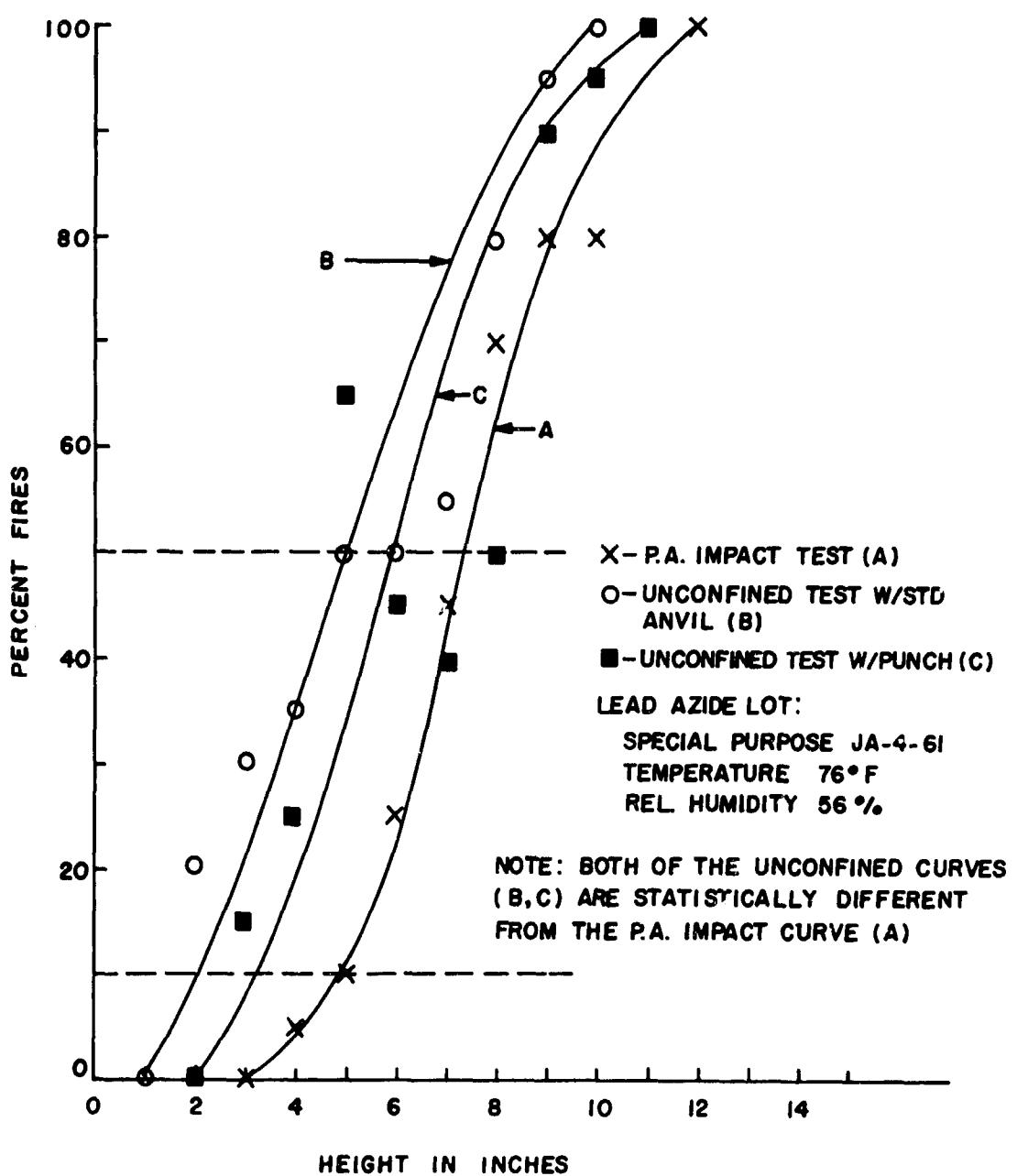


Fig 6 Comparison of impact test methods for lead azide, 2 kg drop weight

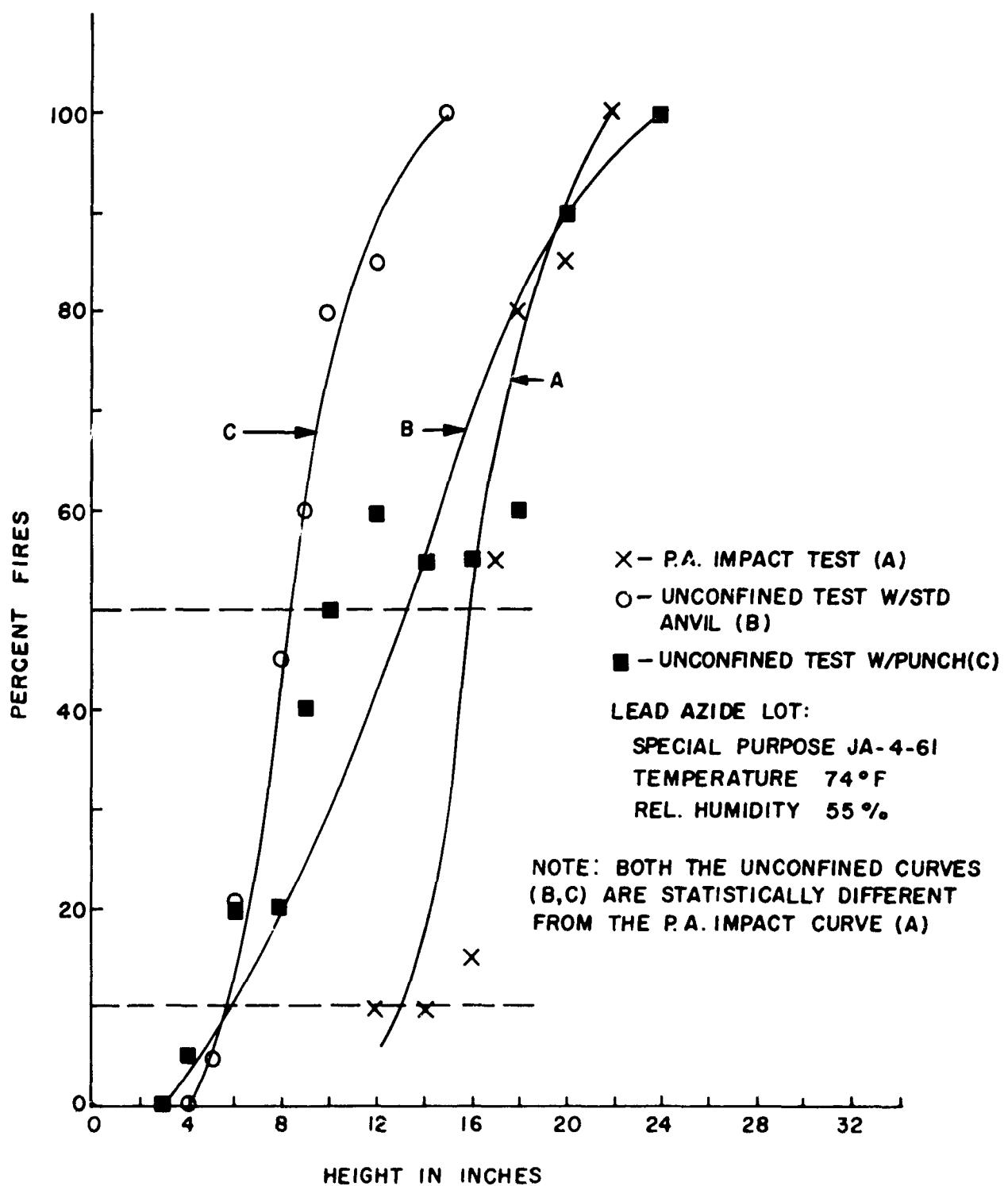


Fig 7 Comparison of impact test methods for lead azide, 1 kg drop weight

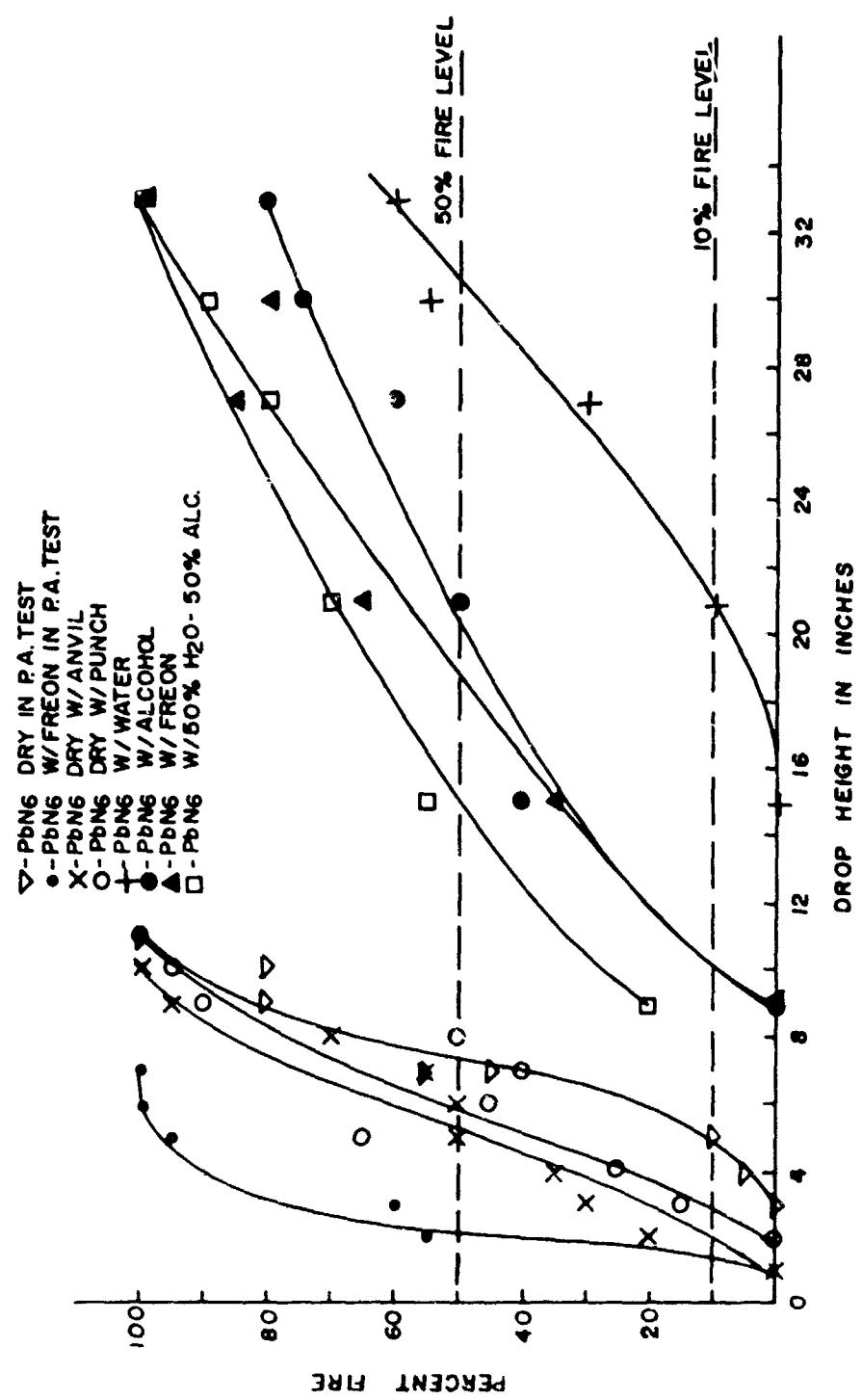


Fig 8 Comparisons of Picatinny Arsenal standard and unconfined impact tests of lead azide and lead azide in well filled with different liquids.
 Note: Unconfined test used 1 3/4" D x 1" H well in steel cylinder with guide for 3/8" punch. Punch resting on explosive. Well filled with liquid. 2-kg drop weight

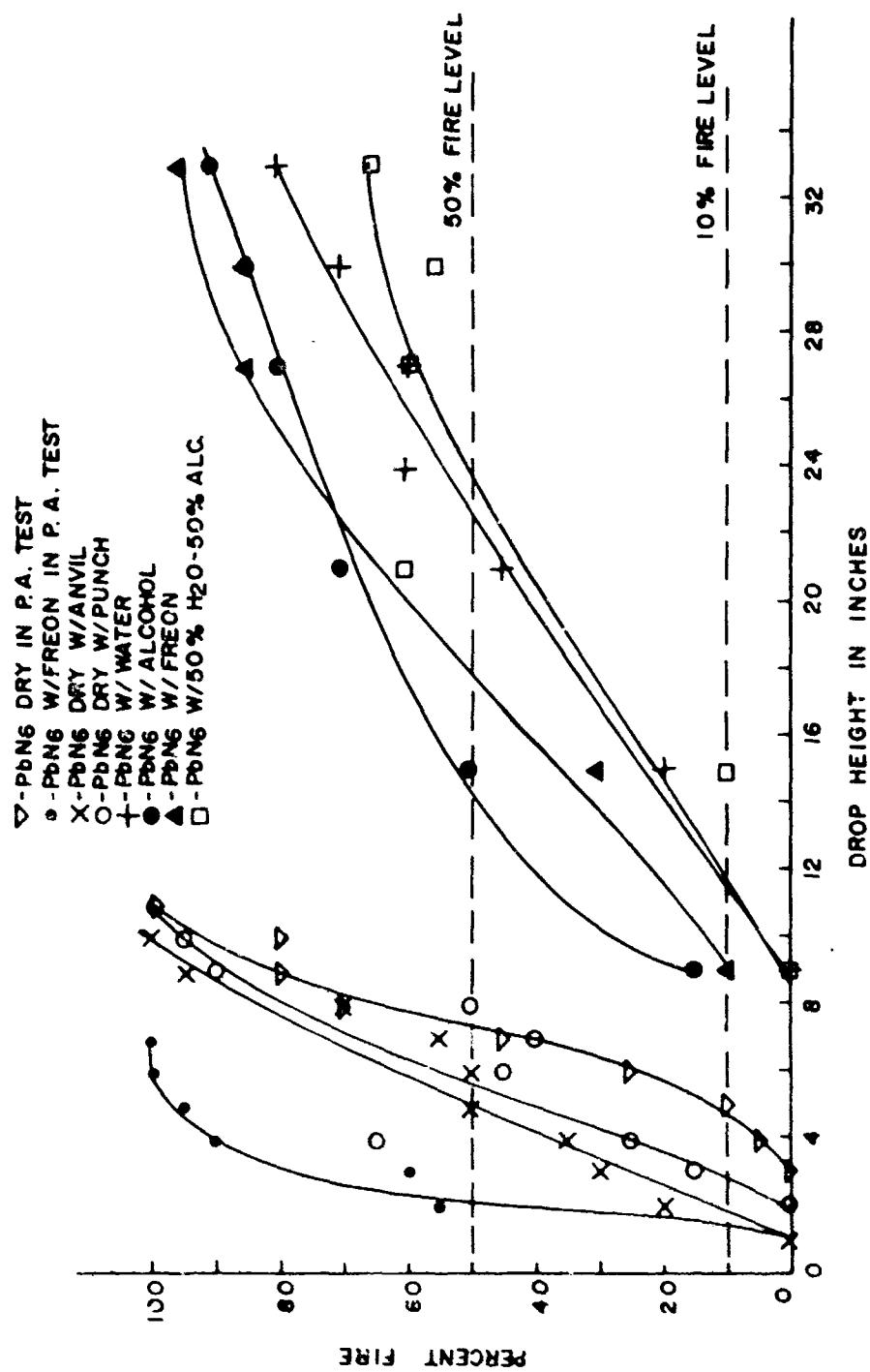


Fig 9 Comparisons of Picatinny Arsenal standard and unconfined impact tests of lead azide and lead azide in well partially filled with different liquids. Note: Unconfined test used 1 3/4" D x 1" H well in steel cylinder with guide for 3/8" punch. Punch resting on explosive with level of liquid just above height of explosive. 2-kg drop weight

UNCLASSIFIED

Security Classification

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11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY
13. ABSTRACT		
<p>The work described in this report was conducted to determine the sensitivity of unconfined mixtures of lead azide with various liquids as measured with an impact machine. The test was developed by so modifying the standard Picatinny Arsenal impact test that the confinement of the sample was reduced; a well was introduced to retain the sample in relatively large amounts of liquid during impact.</p> <p>The results indicate that dry, unconfined lead azide is initiated by impact at lower drop heights than the same material confined in the standard impact-test fixture. The reason for this apparent increase in impact sensitivity is not clear: it may be attributed to some uncontrolled experimental parameter. Unconfined mixtures of lead azide and water, lead azide and alcohol, lead azide and water-alcohol, and lead azide and Freon TF were found to be less sensitive to impact than dry lead azide.</p>		

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Impact sensitivity Lead azide Impact test Freon TF Karber test Goodness-of-fit test Drop height						

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